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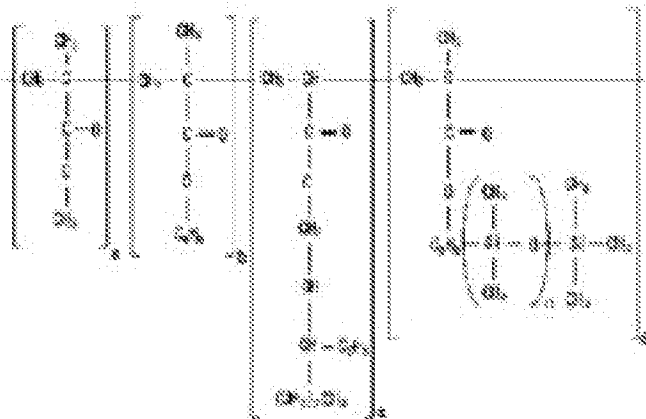
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(21)Application **2000-** (71) **DAITO**  
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**(54) PIGMENT FOR COSMETIC AND COSMETIC  
CONTAINING THE PIGMENT**

(57)Abstract:  
**PROBLEM TO BE  
SOLVED:** To  
provide a cosmetic  
pigment which  
permits the  
employment of a  
generally used oil, a  
generally used  
surfactant, and so on,  
can give a moist  
touch and has an



excellent water-repelling and oil-repelling property, and to provide a cosmetic containing the cosmetic pigment.

SOLUTION: This pigment for

cosmetics, characterized by simultaneously treating the surface of a powdery pigment for the cosmetics with a perfluoroalkyl group-having phosphate represented by the general formula (1) [ $R_f$  is a 3 to 21C perfluoroalkyl or a perfluorooxyalkyl which may be linear or branched and may have a single chain length or plural chain lengths; (n) is an integer of 1 to 12; (y) is the number of 1 to 3; M is H, an alkali metal, an ammonium or a substituted ammonium] and with an ester which is represented by the general formula (2) [(n) is an integer; (a), (b), (c) and (d) are molar ratios, respectively, in the polymer, provided that (a), (b), (c) and (d) are not each zero; (d) is 40 to 60 mol.%] and is obtained by esterifying a copolymer with methylpolysiloxane whose methyl groups are partially substituted by hydroxypropyl, wherein the copolymer has a mol.wt. of 30,000 to 300,000 and is obtained by copolymerizing 2-ethylhexyl acrylate, methacrylic acid, methyl methacrylate, and butyl methacrylate.

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention]This invention relates to the cosmetic containing the paints for cosmetics in which it is blended with the charge of face make up or sun screen cosmetics, such as foundation, eye shadow, and cheek red, for example, and water and oil repellency is shown, and its paints for cosmetics.

[0002]

[Description of the Prior Art]In the paints for cosmetics conventionally used for the charge of face make up or sun screen cosmetics, such as foundation, eye shadow, and cheek red, etc., If there is no water repellence, it will be easy to cause messy makeup by water quality, such as perspiration of the skin, a tear, and rain, and if there is no oil repellency, it will be easy to cause messy makeup by the lipid of the skin, etc.

[0003]Then, in JP,H5-86984,B, JP,H3-246210,A, JP,H4-330007,A, etc. for example, What gave water and oil repellency to the paints is proposed by carrying out the surface treatment of the surface of paints with a perfluoro alkyl phosphate compound.

[0004]However, even if it uses this perfluoro alkyl phosphate compound, Only with what is called the oil of the aliphatic series system generally used or a silicon system, and a binder and a surface-active agent. From a satisfying cosmetic with

a good feel not being obtained by a good dispersion state, as indicated by JP,H4-224506,A and JP,H5-39209,A, for example, Using the oils of fluorine systems, such as perfluoro polyether, a perfluoro decalin, and perfluoro octane, is performed.

[0005]

[Problem to be solved by the invention]However, since the oils of fluorine systems, such as the above-mentioned perfluoro polyether, a perfluoro decalin, and perfluoro octane, were very expensive, combining this had the problem of being restricted only to a specific cosmetic. Foundation also especially had the problem of being a chalky feel, when changing the cosmetic obtained using these oils, the feel entirely made very light, and the way of speaking.

[0006]The purpose of this invention is as follows.

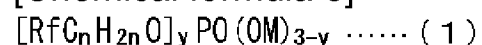
Oil, a surface-active agent, etc. which were made in order to cancel such a problem, and are generally used can be used.

Give a wet feel and provide the cosmetic containing the paints for cosmetics which were moreover excellent in water and oil repellency, and its paints for cosmetics.

[0007]

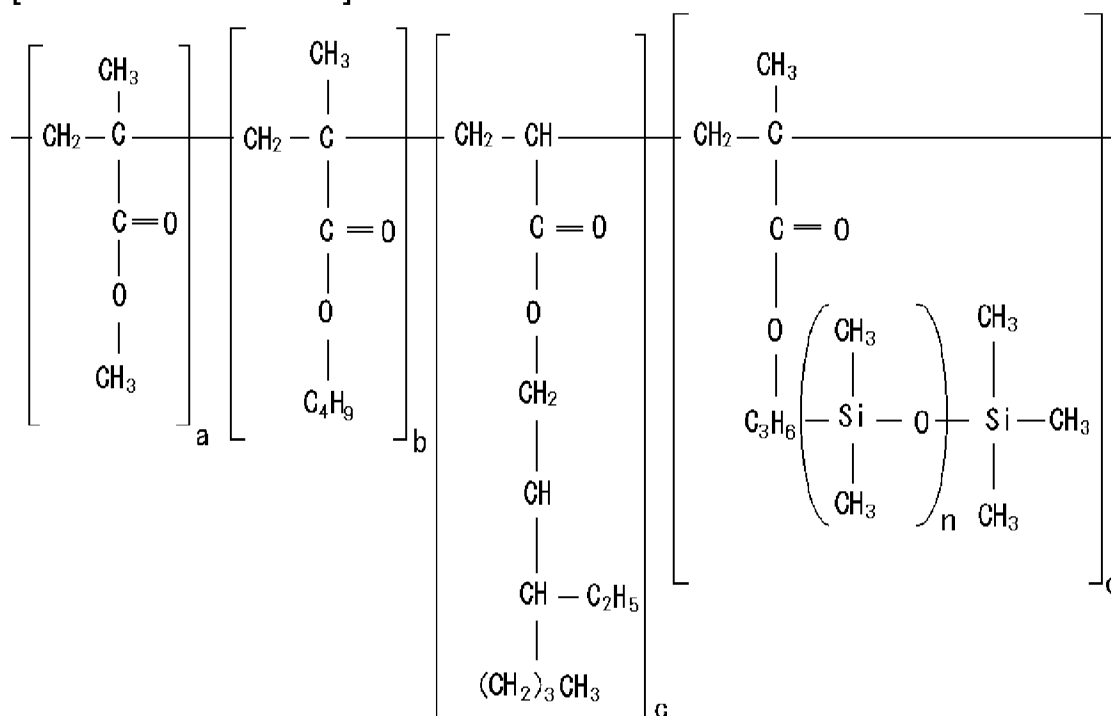
[Means for Solving the Problem and its Function and Effect]In order to attain said purpose, paints for cosmetics by the 1st invention, Phosphoric ester which has a perfluoroalkyl group shown in a paints granular material for cosmetics with a following general formula (1), A molecular weight shown with a following general formula (2) 2-ethylhexyl acrylate of 30,000-300,000, The surface treatment of the ester of what replaced a part of methacrylic acid, methyl methacrylate, and a copolymer of butyl methacrylate and a methyl group of a methylpolisiloxane with a hydroxypropyl group is carried out simultaneously.

[Chemical formula 3]



(式中、Rfは炭素数3～21のパーフルオロアルキル基または、パーフルオロオキシアルキル基を示し、直鎖状あるいは分岐状であって、単一鎖長のものであっても複合鎖長のものであってもよい。nは1～12の整数を示し、yは1～3の数を示す。Mは水素、アルカリ金属、アンモニウムまたは置換アンモニウムを示す。)

[Chemical formula 4]



.....( 2 )

(式中、nは整数で、a, b, c, dは共重合体内のそれぞれのモル比であり、0であることはなく、dは、40モル%以上で60モル%以下である。)

[0008]. While having water and oil repellency according to the paints for cosmetics of this invention, generally are used. For example, vaseline, a ceresin, microcrystallin wax, Kalna Barrow, a candelilla low, higher fatty acid, higher alcohol, squalane, a liquid paraffin, ester oil, and a jig resaler -- a common surface-active agent etc. can be used with oil, such as ide, triglyceride, and silicone oil. When it blends as charges of face make up and sun screen cosmetics, such as foundation, eye shadow, and cheek red, it can be made to distribute and blend easily, and when it is used as a cosmetic, a feel gently used as skin can be given.

[0009]In this invention, it is preferred that coating volume of ester which coating volume of phosphoric ester shown by said general formula (1) is made into 30 or less weight %, and is shown by said general formula (2) is made into 0.1 to 10 weight % (the 2nd invention). Although coating volume of phosphoric ester

shown by said general formula (1) changes with particle diameter of raw material powder covered here and it should be determined in a place which shows sufficient water and oil repellency, Since it may have bad influence on a feel when the compound of a general formula (1) itself comes to deposit and it blends as a cosmetic if the coating volume generally exceeds 30 weight %, it is good to carry out to 30 or less weight %. Although it changes with particle diameter of raw material powder covered on the other hand also about coating volume of ester shown by said general formula (2), generally the target granular material will be obtained with 0.1 to 10weight % of coating volume. When it blends as a cosmetic that this coating volume is less than 0.1 weight %, when good distribution is not obtained or it blends with a cosmetic, with an oil binder generally used, admiration may not be obtained gently. If this coating volume exceeds 10 weight %, oil repellency from which covering by a general formula (1) is also hung down may stop being fully revealed, copolymer of a general formula (2) may serve as a binder, and may put granular material particles together, and it may become a bad granular material of a feel.

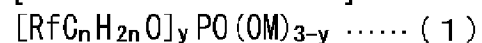
[0010]Next, the 3rd invention relates to a cosmetic containing paints for cosmetics of said 1st invention or the 2nd invention. According to the cosmetic of this invention, it has the function for water and oil repellency to be shown and to prevent messy makeup, and a cosmetic where admiration was moreover given gently and which was excellent in a using feeling can be obtained.

[0011]

[Mode for carrying out the invention]Next, an embodiment of a cosmetic containing paints for cosmetics by this invention and its paints is described.

[0012]In paints for cosmetics by this invention, it is a following general formula (1).;

[Chemical formula 5]

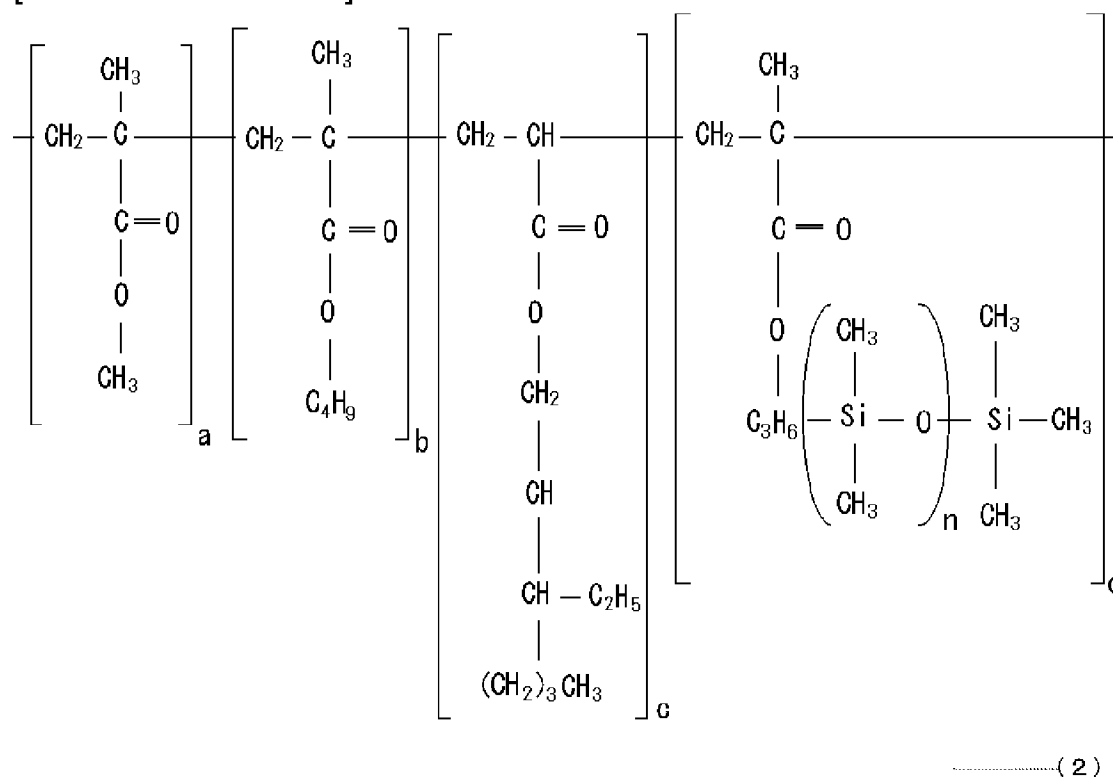


(式中、Rfは炭素数3～21のパーフルオロアルキル基または、パーフルオロオキシアルキル基を示し、直鎖状あるいは分岐状であって、単一鎖長のものであっても複合鎖長のものであってもよい。nは1～12の整数を示し、yは1～3の数を示す。Mは水素、アルカリ金属、アンモニウムまたは置換アンモニウムを示す。)

As phosphoric ester which has a perfluoroalkyl group shown by being alike, For example, a perfluoro alkyl-phosphoric-acid ester diethanolamine salt which is the solid content of a moisture powder emulsion marketed under the name of AG-530 from Asahi Glass Co., Ltd., Or although it has the same molecular structure as this, a perfluoro alkyl-phosphoric-acid ester sodium salt, perfluoro alkyl-phosphoric-acid ester potassium salt, perfluoro alkyl-phosphoric-acid ammonium salt, etc. in which forms of a salt differ are mentioned.

[0013]On the other hand, it is a following general formula (2).;

[Chemical formula 6]



(式中、nは整数で、a, b, c, dは共重合体内のそれぞれのモル比であり、0であることはなく、dは、40モル%以上で60モル%以下である。)

As a copolymerization compound ("acrylic silicon copolymer" is called hereafter.) shown by being alike, For example, copolymer dissolved in isopropanol, butyl acetate, and volatile silicon which are marketed under the name of KP-541, KP-543, and KP-545 from Shin-Etsu Chemical Co., Ltd. is mentioned.

[0014]Methods of covering simultaneously these perfluoro alkyl phosphate compound and acrylic silicon copolymer on the surface of a paints granular

material for cosmetics include following methods.

[0015]First, a perfluoro alkyl phosphate compound and acrylic silicon copolymer of the specified quantity which should be covered by the surface so that concentration may be about 0.5 to 20 wt% as a solid, respectively, a suitable solvent -- simultaneous -- the dissolution -- or the distributed emulsion is formed, a paint granular material for cosmetics is added during this heating and stirring, and fixed time churning is carried out. Then, after heating (it decompresses in being required) and removing a solvent, it heats at about 110 °C for 6 hours, and a paint granular material for water-and-oil-repellency cosmetics which is ground after that and made into the purpose is obtained. As a solvent used here, organic solvents, such as isopropyl alcohol, butyl acetate, volatile silicon, and acetone, are preferred.

[0016]In this way, phosphoric ester which has a perfluoroalkyl group shown in a paint granular material for water-and-oil-repellency cosmetics obtained by said general formula (1), and acrylic silicon copolymer shown by said general formula (2) are simultaneously covered with a surface treatment.

[0017]Here, although coating volume in particular of phosphoric ester shown by said general formula (1) is not limited, it should just be the minimum coating volume from which it changes with particle diameter of a paint granular material for cosmetics used as a raw material, and sufficient water and oil repellency is obtained. However, if the coating volume generally exceeds 30 weight %, the compound of a general formula (1) itself comes to deposit, and when it blends as a cosmetic, it may have bad influence on a feel.

[0018]On the other hand, although not limited in particular for coating volume of ester shown by said general formula (2), generally 0.1 to 10 weight % of coating volume is suitable. When it blends as a cosmetic that this coating volume is less than 0.1 weight %, an oil binder (for example, vaseline, a ceresin, and microcrystalline wax.) generally used Kalna Barrow, a candelilla low, higher fatty acid, higher alcohol, squalane, a liquid paraffin, ester oil, and a jig resaler -- with ide, triglyceride, silicone oil, etc. A good cosmetic of a dispersion state cannot be obtained with a common surface-active agent etc., When a case where oils of fluorine systems, such as perfluoro polyether, a perfluoro decalin, and perfluoro



octane, must be used arises or it blends with a cosmetic, admiration may not be obtained gently. If this coating volume exceeds 10 weight %, oil repellency from which covering by a general formula (1) is also hung down may stop being fully revealed, acrylic silicon copolymer of a general formula (2) may serve as a binder, and may put granular material particles together, and it may become a bad granular material of a feel.

[0019]As paints for cosmetics in which surface coating is carried out by this invention, there are an inorganic pigment, an organic color, and resin powder paints. As an inorganic pigment here Titanium oxide, red ocher, yellow oxide of iron, black iron oxide, Ultramarine, a flower of zinc, magnesium oxide, zirconium oxide, mica, a sericite, talc, silica, kaolin, calcium carbonate, chromium hydroxide, a magnesium silicate, an aluminum oxide, barium sulfate, titanium covering mica, etc. are mentioned. As an organic color, the litholrubin B, Lake Red C, Lithol Red, rhodamine B, the HERIN boss pink CN, Permanent Red, the benzidine orange G, copper phthalocyanine blue, etc. are mentioned. As resin powder paints, nylon powder, acrylic powder, silicon powder, etc. are mentioned.

[0020]When blended with a cosmetic according to the paints for cosmetics of this invention, perfluoro polyether of a fluorine system, Without using a disperse composition agent of fluorine systems, such as a perfluoro decalin and perfluoro octane, As a distributed oily component, for example, vaseline, a ceresin, microcrystallin wax, Kalna Barrow, a candelilla low, higher fatty acid, higher alcohol, squalane, a liquid paraffin, ester oil, and a jig resaler -- combination adjustment can be easily carried out at a cosmetic using general oily components, such as ide, triglyceride, and silicone oil. In this cosmetic by which combination adjustment was carried out, a feel which showed sufficient water and oil repellency which should be demanded, and was gently used as skin can be given.

[0021]As loadings of said paints for cosmetics blended with a cosmetic, 0.1 to 100 weight % is preferred to a total amount of a cosmetic, and it is 1 to 60 weight % more preferably. As a pharmaceutical form of said cosmetic, conventionally publicly known pharmaceutical forms, such as the shape of a bilayer, a water-in-oil type emulsion, an oil-in-water type emulsion, gel form, a spray, the shape of a

mousse, oiliness, and a solid state, can be used. For a use of a sun block, the shape of a bilayer, a water-in-oil type emulsion, and gel form are preferred especially, and a solid state, a solid emulsion, gel form, a water-in-oil type emulsion, an oil-in-water type emulsion, oil, the shape of a mousse, etc. are preferred for a use of foundation.

[0022]

[Working example]Next, a concrete embodiment of a cosmetic containing paints for cosmetics by this invention and its paints is described.

[0023](Manufacture embodiment 1) A carbon number makes it dissolve in isopropyl alcohol with a number average, so that solid content of an acrylic silicon copolymer (Shin-Etsu Chemical KP-541) may be 1wt% 3.5wt% as solid content in perfluoro alkyl-phosphoric-acid ester ammonium salt of 13. At this time, perfluoro alkyl-phosphoric-acid ester ammonium salt existed as an emulsion in isopropyl alcohol, without dissolving thoroughly, and a solution is cloudy.

[0024]Next, pour 5-kg paints (titanium oxide) into a high-speed agitation mixing machine, and 10 kg of the above-mentioned solutions are dropped during this agitation mix, After having agitated at 80 °C for 30 minutes, having warmed at 120 °C after that, having decompressed the agitation mixing inside of a plane, maintaining for about 2 hours and removing thoroughly isopropyl alcohol which is a solvent, it heat-treated at 110 °C for 6 hours.

[0025]Subsequently, after paying out of the agitation mix inside of a plane, the hammermill into which a 2-mm screen was put ground, and 7wt% of the carbon number carried out surface coating to perfluoro alkyl-phosphoric-acid ester of 13 by 2wt% of acrylic silicon copolymer.

[0026]A total of seven kinds of paints for cosmetics which were replaced with titanium oxide as paints and made into the same amount of surface coating in the similar way using a sericite, mica, talc, yellow oxide of iron, red ocher, and black iron oxide were obtained.

[0027](Manufacture embodiment 2) A carbon number makes it dissolve in isopropyl alcohol with a number average, so that the solid content of an acrylic silicon copolymer (Shin-Etsu Chemical KP-541) may be 2wt% 4wt% as solid

content in perfluoro alkyl-phosphoric-acid ester potassium salt of 13. About except [ this ], the titanium oxide in which 8wt% of the carbon number carried out surface coating to perfluoro alkyl-phosphoric-acid ester of 13 by 4wt% of acrylic silicon copolymer was obtained by the same method as the manufacture embodiment 1.

[0028]A total of seven kinds of paints for cosmetics which were replaced with titanium oxide as paints and made into the same amount of surface coating in the similar way using a sericite, mica, talc, yellow oxide of iron, red ocher, and black iron oxide were obtained.

[0029](Manufacture comparative example 1) Although the manufacturing method is the same as that of the manufacture embodiment 1, The thing which made isopropyl alcohol emulsion-ize only perfluoro alkyl-phosphoric-acid ester ammonium salt of 3.5wt% of the carbon number 13 is used, The paints which carried out surface coating to said each paints (titanium oxide, a sericite, mica, talc, yellow oxide of iron, red ocher, black iron oxide) with perfluoro alkyl-phosphoric-acid ester of 7wt% of the carbon number 13 were obtained.

[0030](Manufacture comparative example 2) Although the manufacturing method is the same as that of the manufacture embodiment 1, To isopropyl alcohol, perfluoro alkyl-phosphoric-acid ester potassium salt of 4wt% of the carbon number 13, The solution in which 7wt% of the acrylic silicon copolymer (Shin-Etsu Chemical KP-541) was dissolved by solid content is used, The paints which carried out surface coating to said each paints (titanium oxide, a sericite, mica, talc, yellow oxide of iron, red ocher, black iron oxide) by both perfluoro alkyl-phosphoric-acid ester of 8wt% of the carbon number 13 and 14wt% of acrylic silicon copolymer were obtained.

[0031]Water repellency and oil-repellent degree were measured about each surface coating paints of these manufacture embodiments 1 and 2 and the manufacture comparative examples 1 and 2. This measurement result is shown in Table 1. Here, about water repellency, an angle of contact according an angle of contact by water to paraffin about oil-repellent degree was measured. The Elmer goniometer type angle-of-contact measuring device was used for measurement of this angle of contact.

[0032]

[Table 1]

顔料種類	製造実施例 1		製造実施例 2		製造比較例 1		製造比較例 2	
	撥水度	撥油度	撥水度	撥油度	撥水度	撥油度	撥水度	撥油度
酸化チタン	152	132	155	131	150	129	142	101
セリサイト	141	122	145	124	140	120	140	90
マイカ	140	124	141	121	138	119	132	85
タルク	138	47	140	50	135	41	88	86
黄酸化鉄	153	135	155	131	150	132	148	109
ベンガラ	154	136	154	135	154	133	142	104
黒色酸化鉄	150	138	152	130	149	134	149	110

[0033](Embodiments 1 and 2) Powder foundation was prepared by the following combination using paints prepared in manufacture embodiments 1 and 2, respectively.

Surface coating sericite 35.0 surface-coating talc 25.0 surface-coating mica 20.0  
 Surface coating titanium oxide 10.0 surface-coating yellow oxide of iron 4.0  
 surface-coating red ochre 1.2 surface-coating black iron oxide 0.8 nylon-powder

4.0100.0[0034]It contained 88% by having used said powder foundation as a powder base, a binder base was made into 12%, and these were mixed with a Henschel mixer. At this time, the combination of a binder base is as follows.

Dimethylpolysiloxane (6CS) 30 dimethylpolysiloxane (10000CS) 25 purified-lanolin 9 ester-oil 36100[0035](Embodiments 3 and 4) W/O facial liquid

foundation was prepared by the following combination using the paints prepared in manufacture embodiments 1 and 2, respectively.

Surface coating talc 3.0 Surface coating sericite . 2.0 Surface coating titanium oxide 15.0 surface-coating red ochre 0.2 surface-coating yellow oxide of iron 2.5 surface-coating black iron oxide 0.8 methylpolysiloxane (6CS) 7.4

Dimethylcyclohexanemethyl (polyoxyethylene)

Dimethylsiloxane copolymer 1.8 Methylpolysiloxane (100CS) 8.0

Methylpolysiloxane (10000CS) 4.0 methylphenyl polysiloxane 10.0 Ethanol 10.0 1.3-butylene-glycol 2.0 purified water 33.3100.0[0036](Comparative examples 1

and 2) Powder foundation was prepared by the same combination as

Embodiments 1 and 2 using the paints prepared by the manufacture comparative examples 1 and 2 as surface coating paints.

[0037](Comparative examples 3 and 4) W/O facial liquid foundation was prepared by the same combination as Embodiments 3 and 4 using the paints prepared by the manufacture comparative examples 1 and 2 as surface coating paints.

[0038]About each cosmetic of these Embodiments 1-4 and the comparative examples 1-4, a dispersion state, state stability, and a water-repellent and oil-repellent result were summarized. This result is shown in Table 2.

[0039]

[Table 2]

	分散状態	状態安定性	撥水性	撥油性
実施例 1	◎	◎	○	○
実施例 2	◎	◎	◎	◎
実施例 3	○	◎	○	○
実施例 4	○	◎	○	○
比較例 1	△	ケーキング	○	○
比較例 2	○	○	○	×
比較例 3	×	×	—	—
比較例 4	○	○	○	×

[0040]According to the surface coating paints prepared in manufacture embodiments 1 and 2, satisfying water-repellent oil repellency is obtained from the result shown in the above-mentioned table 1. On the other hand, although water and oil repellency sufficient in the manufacture comparative example 1 is shown, the manufacture comparative example 2 is enough as water repellence, but oil repellency serves as a little low value.

[0041]From the result shown in Table 2, according to the powder foundation of Embodiments 1 and 2, the dispersion state was also good, good water and oil repellency was shown, and the state is stable, without starting caking. The using feeling is carried out gently and did not become a chalky feel. On the other hand, in the powder foundation of the comparative example 1, although good water and oil repellency is shown, caking is started and it cannot be said to be good powder

foundation. In the powder foundation of the comparative example 2, dispersibility is good, caking is not started, and although state stability is also good, sufficient oil repellency is not shown.

[0042]On the other hand, in the W/O facial liquid foundation of Embodiments 3 and 4, the stability of a W/O emulsion and a dispersion state are also good, and the feel which showed good water and oil repellency when it applied to skin, and was carried out gently is given. On the other hand, in the comparative example 3, a W/O emulsion could not be very unstable, separation was not able to be caused immediately, and stable W/O facial liquid foundation was not able to be obtained. According to the comparative example 4, although it is easy to form a stable W/O emulsion and a cosmetic is made, when it applies to skin, good oil repellency is not shown.

[0043]As mentioned above, while water and oil repellency is excellent according to the cosmetic of this example which blended the paints for cosmetics and its paints for cosmetics of this manufacture embodiment, At the time of combination of a cosmetic, perfluoro polyether, a perfluoro decalin, Without blending the binder of fluorine compound systems, such as perfluoro octane, the disperse composition agent (for example, vaseline, a ceresin, and microcrystallin wax.) generally used Kalna Barrow, a candelilla low, higher fatty acid, higher alcohol, squalane, a liquid paraffin, ester oil, and a jig resaler -- it can be said that it has the outstanding effect that disperse composition can be easily carried out with ide, triglyceride, silicone oil, etc.

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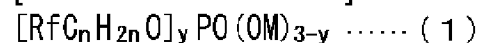
## CLAIMS

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[Claim(s)]

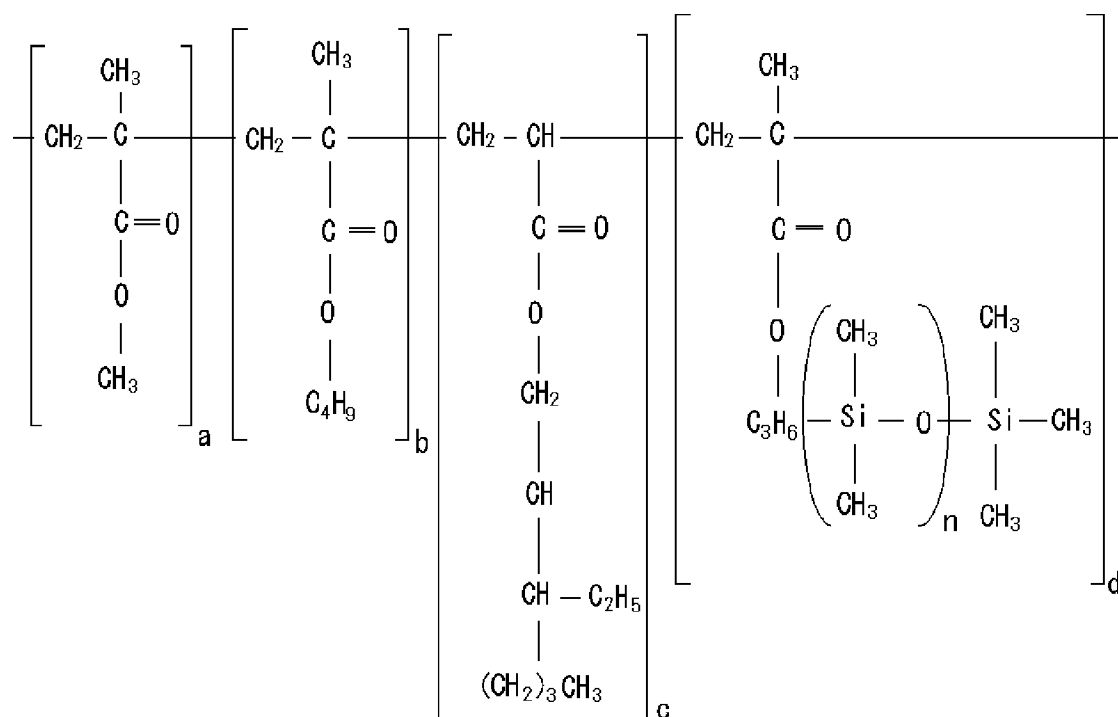
[Claim 1]Phosphoric ester which has a perfluoroalkyl group shown in a paints granular material for cosmetics with a following general formula (1), A molecular weight shown with a following general formula (2) 2-ethylhexyl acrylate of 30,000-300,000, Paints for cosmetics which carry out the surface treatment of the ester of what replaced a part of methacrylic acid, methyl methacrylate, and a copolymer of butyl methacrylate and a methyl group of a methylpolisiloxane with a hydroxypropyl group simultaneously, and are characterized by things.

[Chemical formula 1]



(式中、Rfは炭素数3～21のパーフルオロアルキル基または、パーフルオロオキシアルキル基を示し、直鎖状あるいは分岐状であって、単一鎖長のものであっても複合鎖長のものであってもよい。nは1～12の整数を示し、yは1～3の数を示す。Mは水素、アルカリ金属、アンモニウムまたは置換アンモニウムを示す。)

[Chemical formula 2]



.....(2)

(式中、nは整数で、a, b, c, dは共重合体内のそれぞれのモル比であり、0であることはなく、dは、40モル%以上で60モル%以下である。)

[Claim 2]The paints for cosmetics according to claim 1 with which coating volume of ester which coating volume of phosphoric ester shown by said general formula (1) is made into 30 or less weight %, and is shown by said general formula (2) is made into 0.1 to 10 weight %.

[Claim 3]A cosmetic which contains said paints for cosmetics according to claim 1 or 2, and is characterized by things.

[Translation done.]